

## \* NOTICES \*

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- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

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DETAILED DESCRIPTION

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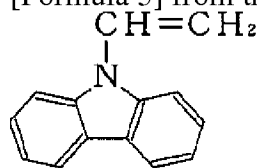
[Detailed Description of the Invention]

[0001]

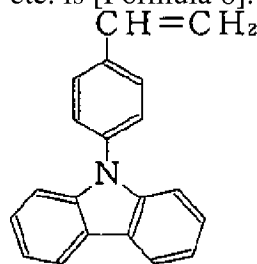
[Field of the Invention] This invention relates to those synthetic approaches at the new monomer which consists of a new carbazole derivative, and a new polymer list.

[0002]

[Description of the Prior Art] As an example of the vinyl monomer as a carbazole derivative, it is [Formula 5] from the former.



The becoming vinylcarbazole and its polymer are known and there is reference indicated that the polymer is used for an organic electroluminescence (EL) component. Moreover, this invention person etc. is [Formula 6].



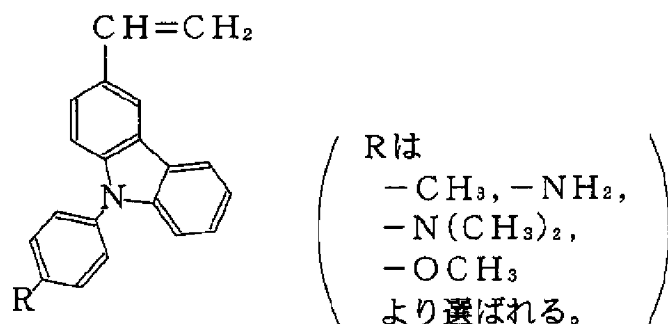
It indicates to those manufacture approaches at becoming 4-carbazole styrene and its polymer, and a pan, and the application to organic electroluminescence is indicated in the list at Japanese Patent Application No. No. 260328 [ ten to ].

[0003]

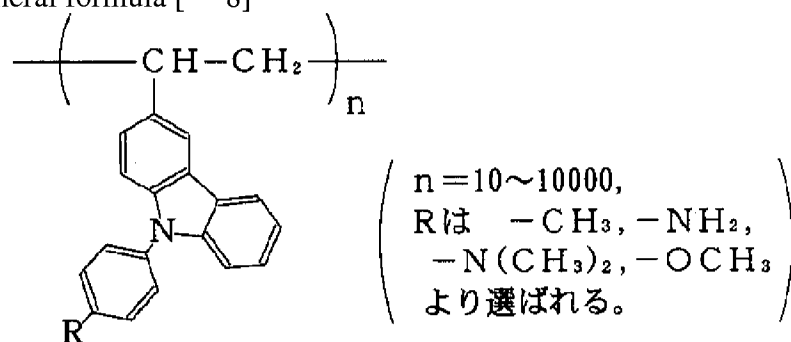
[Problem(s) to be Solved by the Invention] this invention persons aim at discovery and its manufacture approach of the carbazole derivative which is not in the former from which the application to an organic EL device is expected.

[0004]

[Means for Solving the Problem] In view of said purpose, wholeheartedly, it is found out and this invention is a general formula [\*\* 7] after research.



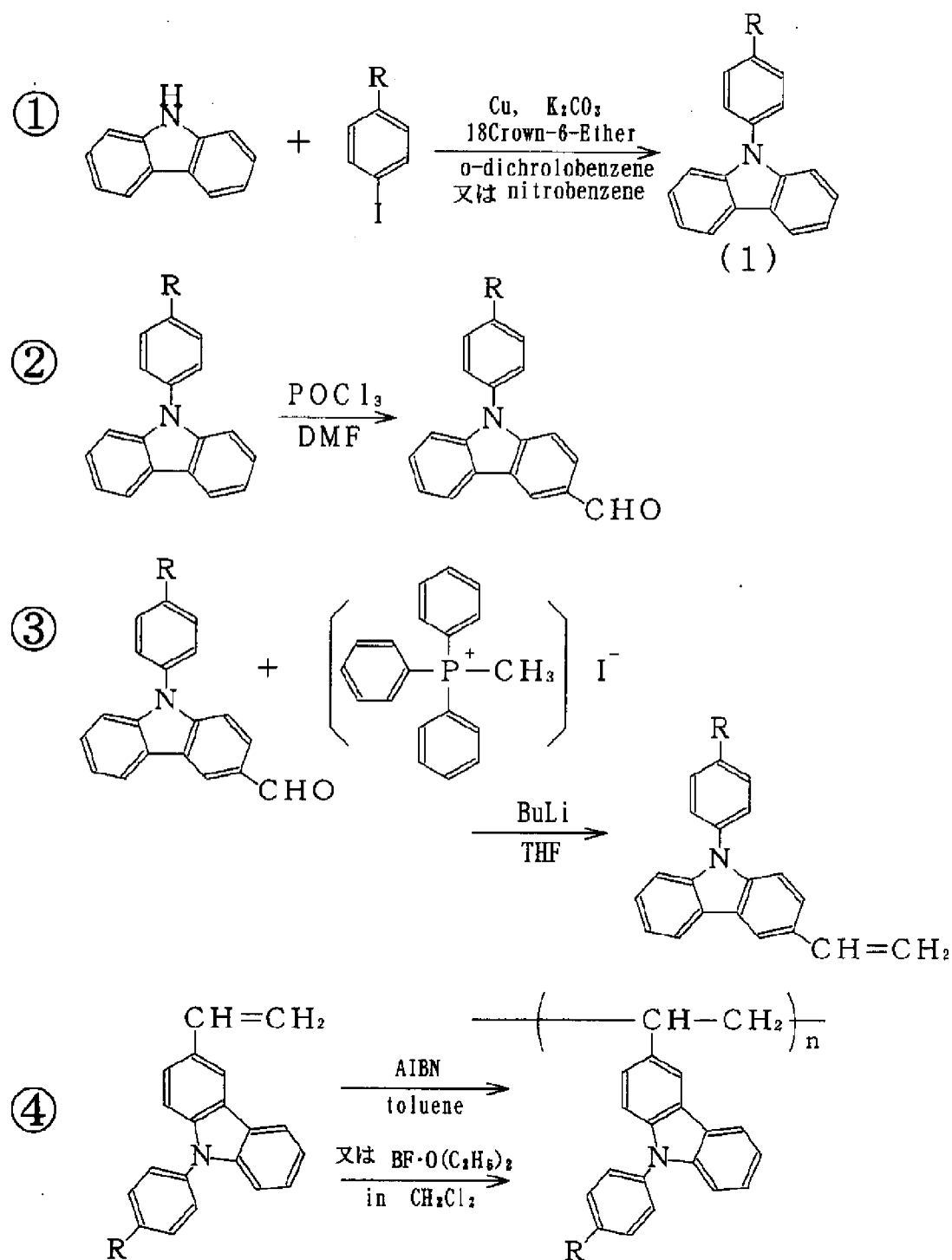
The new monomer (monomer) which comes out and consists of a carbazole derivative shown, and a general formula [\*\* 8]



It comes out and is related with the new polymer (polymer) shown.

[0005] Moreover, this invention also proposes the synthetic approach of of the monomer and polymer which are said carbazole derivative, and these are compounded through the following processes.

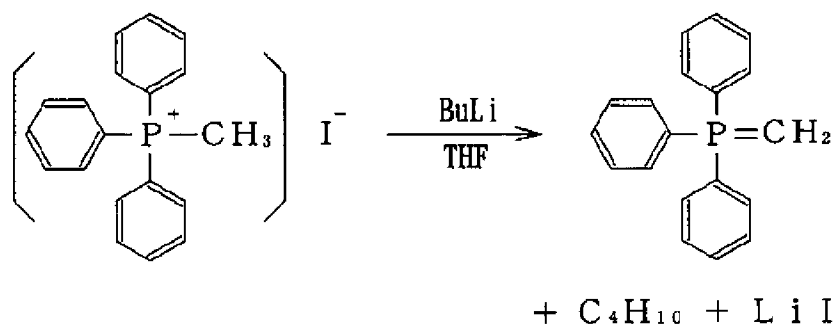
[Formula 9]



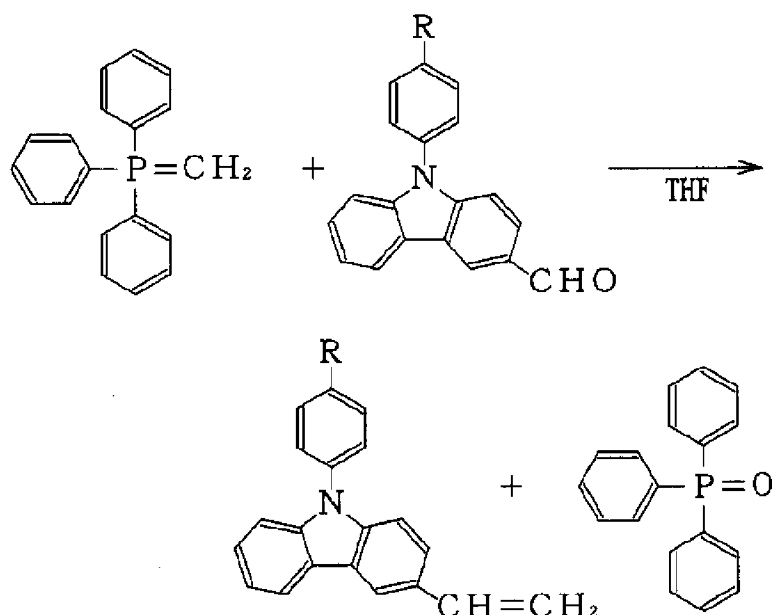
[0006]

[Embodiment of the Invention] A carbazole and an iodine compound are made to react under said conditions, and a compound (1) is made to compound in the first process [ of said synthetic approach ] \*\*. It is a phosphoryl chloride (POCl<sub>3</sub>) about the compound (1) obtained in the second process \*\*. And N, N - The direct formylation reaction to a ring is performed using dimethylformamide (DMF). At the third process \*\*, in fact, a reaction process is those with two, and [Formula 10], as shown below.

③-1



③-2



A carbazole derivative (monomer) is compounded in a \*\*\*\*\* path. And although the polymerization of this monomer is carried out and a polymer is compounded in the fourth process \*\*, a general polymerization method may be used besides said approach.

[0007] The carbazole derivative monomer obtained at the process of the aforementioned \*\* - \*\* is expected as an application raw material in the field of many besides the application as a raw material of various functional polymer materials. Moreover, the polymer obtained at the process of the aforementioned \*\* is 20000 - 30000 cd/m<sup>2</sup>. Brightness is obtained and it is thought that it is useful also as an ingredient of an organic EL device.

[0008]

[Example] Composition of an example 1 carbazole derivative monomer and its polymerization object

[0009] i. -- first stage story reaction; -- a carbazole and 1-methoxy-4- The reaction mechanical stirrer and bubble tube cooling pipe of an iodobenzene were attached. the inside of a 200ml three-neck flask -- 1-methoxy-4- Iodobenzene 25.0g (0.113 mols) Carbazole 17.9g (0.107 mols) and 18-crown-6-ether 9.7g (0.036 mols), 16.7g (0.12 mol) of potassium carbonate, 1.2g (0.018 mols) of copper In addition, o-dichlorobenzene is added as a solvent and it is N<sub>2</sub>. In the inside of an air current, and a silicone oil bath

It heats, and it agitated for 24 hours and was made to react to 195 degrees C. Suction filtration is carried out using a nutsche with a hot condition, filtrate is covered over the column chromatography in a benzene solvent, after reaction termination, after separation, after condensing by the evaporator, vacuum stoving is carried out, and it is 9. - (4- (1-methoxy) phenyl) Carbazole 13.6g was obtained. Yield was 48.4%.

[0010] ii. second stage story reaction; 9 - ( ) [ 4-(1-methoxy) ] Phenyl and phosphoryl chloride The formylation mechanical stirrer and bubble tube cooling pipe of a carbazole were attached. ( ) They are N and N into a 200ml three-neck flask. - Dimethylformamide (DMF) 7.3g (0.1 mols) [ POC13 ] 15.3 g (0.1 mols) It is 9, agitating continuously, after carrying out mixed churning in a room temperature. - (4- (1-methoxy) phenyl) Carbazole 26.1g (0.1 mols) It dissolved in solvents, such as benzene, added and was made to react at 90 degrees C for 4 hours. In addition to ice-cooling water, benzene extracted generated sediment after reaction termination. After making extract benzene liquid dehydrate by Na<sub>2</sub>SO<sub>4</sub> and carrying out reduced pressure removal of the benzene, it is made to recrystallize in n-hexane, and it is the target 9-(4- (1-methoxy) phenyl)-6-formyl. - Carbazole 21.4g was obtained. Yield was 74%.

[0011] The iii. third staircase reaction; 9-(4- (1-methoxy) phenyl)-6-formyl - Carbazole The reduction vinylation mechanical stirrer and the bubble tube cooling pipe were attached. It is N<sub>2</sub> into a 200ml three-neck flask. 20ml of n-hexane solutions of n-butyl lithium is put in among an air current, using a tetrahydrofuran as a solvent, and it is iodation triphenylmethyl phosphonium under churning. 3.7g was dropped and churning was continued at the room temperature for 4 hours. Subsequently, 2.0 g dissolved in the tetrahydrofuran was made dropped, and it was made to react at a room temperature for 12 hours. Distilled water was made to decompose an unreacted object after reaction termination, and the ether extract was carried out. It carried out by repeating this actuation, washing was terminated in the place where wash water became neutrality, the ether extract was carried out, and it was made to dehydrate by Na<sub>2</sub>SO<sub>4</sub>. Then, the ether is removed and it is target 9-(4- (1-methoxy) phenyl)-6-vinyl. - Carbazole 1.1 g was obtained. Yield was 54.1%.

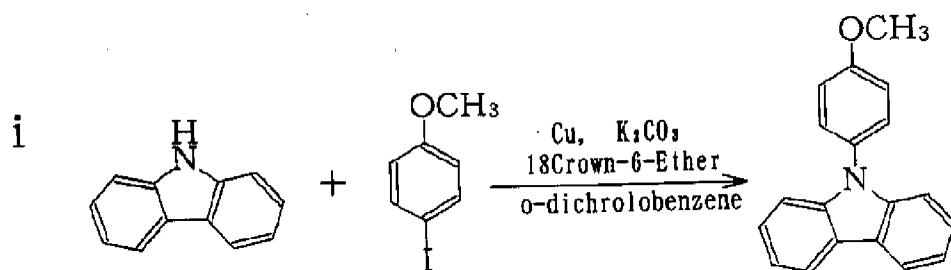
[0012] The iv. fourth staircase reaction;

(The 1) Radical polymerization 9-(4- (1-methoxy) phenyl)-6-vinyl - Carbazole 0.2 g (0.7mmol) It was made to dissolve in toluene 1ml, and azobisisobutyronitrile 2.43mg (0.01mmol) was put into the glass tube as a polymerization initiator. This glass tube was cooled all over the dry ice-methanol bus, and it was made the vacua. After performing a nitrogen purge 3 times and removing oxygen completely, the sealed tube is carried out, and the glass tube was shaken for 20 hours and made to react at 80 degrees C. It returned to the room temperature after reaction termination, and ice-cooled after that. After carrying out an open pipe, the obtained solution was thrown in in the methanol and white precipitate was obtained. It was made to dissolve in dichloromethane and this sediment was settled with the methanol. After repeating this reprecipitation 3 times, polymer 0.09g was obtained. The obtained polymer is m.p.79-91 degree C, and the NMR chart was shown in drawing 1 .

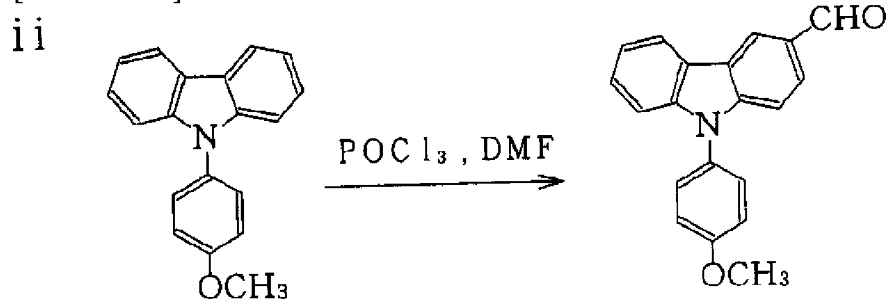
[0013] (The 2) It is 9-(4- (1-methoxy) phenyl)-6-vinyl to the mold flask of 10ml of cationic polymerization to make. - Dichloromethane 2ml was put in as carbazole 0.2 g (0.7mmol) and a solvent. After making the double rubber stopper this mold flask to make and sealing completely, it cooled to -50 degrees C using the dry ice-methanol bus. Very little 3 boron-fluoride-diethylether complex was put into the 1ml hypodermic needle after that, and it thrust into the double rubber stopper, and poured in into the solution. After returning even to a room temperature slowly after that, the solution was thrown in in the methanol and white precipitate was obtained. It was made to dissolve in dichloromethane and this sediment was settled with the methanol. This reprecipitation was acquired for repeat polymer 0.07g 3 times. The obtained polymer was m.p.82-92 degree C.

[0014] If the reaction formula in this synthetic approach is described, it will become as follows.

[Formula 11]

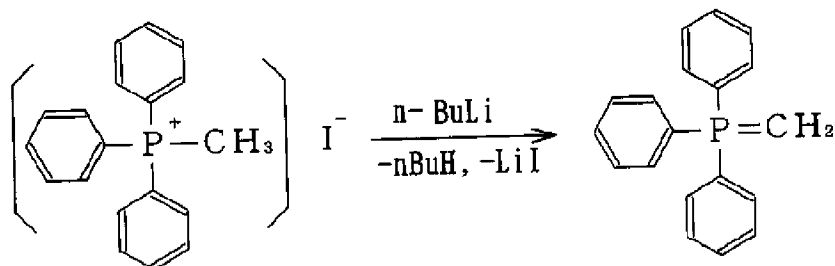


[Formula 12]

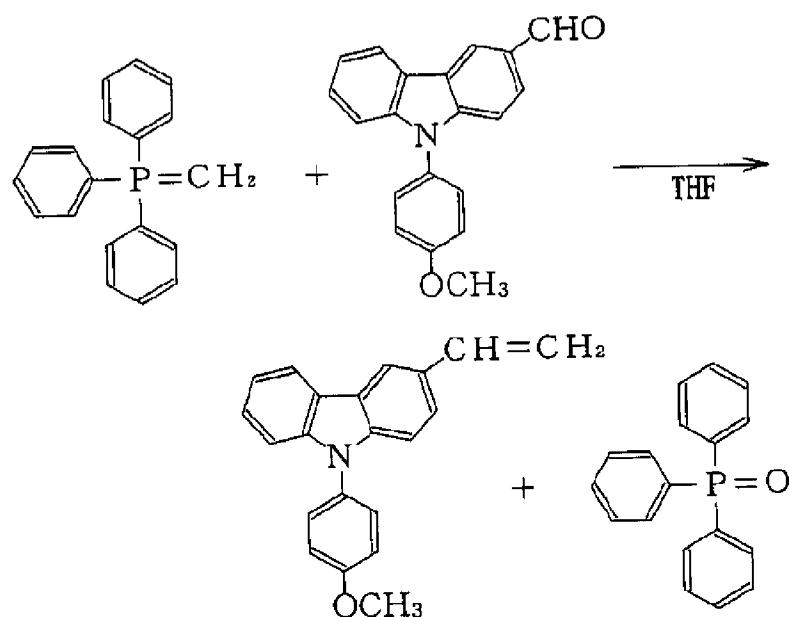


[Formula 13]

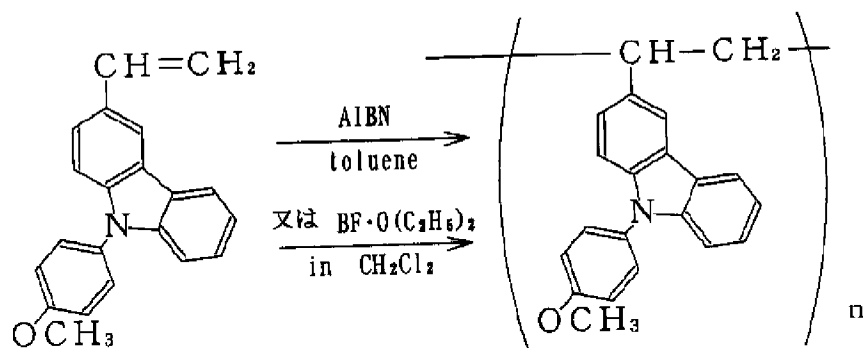
iii-1



iii-2



[Formula 14]



[0015]

[Effect of the Invention] As explained above, the new monomer of this invention can raise thermal resistance compared with the conventional polymer by carrying out copolymerization to other

monomers, such as ethylene, a propylene, a vinyl chloride, and vinyl acetate, and an application temperature region can be increased more to the elevated-temperature section. Moreover, the polymerization object of this invention is excellent in thermal resistance and an electrical property, and can expect further the application including electrical parts, such as various components for automobiles, and lighting fitting, made into a functional polymer, and the use which they are probably in the fields, such as application to the charge of organic EL device material.

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[Translation done.]